#19 3/8/00

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In reapplication of
E. PRAT et al.

Group art Unit : 1754
Examiner : S. HENDRICKSON

Serial N° : CPA of 08/765,901
Filed : January 7, 1997

For : CONCENTRATED SUSPENSION PRECIPITATION SILICA, METHODS OF PREPARATION AND UTILIZATION OF SAID SUSPENSION

DECLARATION UNDER RULE 132

Hon. Commissioner of Patents and Trademarks
WASHINGTON D.C. 20231
Sir :
I, Evelyne PRAT, residing at 20 bis rue jules Auffret 93500 PANTIN, France ;
Declare and say :

I am citizen of France.

I am graduated from Ecole Nationale Supérieure de Chimie de Paris (1983), and I am Doctor in analytical chemistry (1986)

Since 1986, I have been employed by RHONE-POULENC CHIMIE, the Assignee of the pending application, as scientist specialized in the field of silica synthesis.

As a result of my education and professional experience, I consider myself able to interpret what patents or other technical literature would communicate to one of ordinary skill in the art of silica suspensions.

I am the inventor of the present application that specifically aims at providing a silica suspension having a high solid content, a low viscosity and a high stability in time.

I am aware of the fact that US patent No.5,430,570 in the name of Chevallier et al. has been cited in the course of the US procedure as anticipating the subject matter of the pending application.

US 5,403,570 is concerned with providing silica particles, in the form of beads, powder or granulates.

In that respect, US 5,403,570 describes a process comprising the following steps 1) providing a suspension of precipitated silicate, 2) filtering the suspension to obtain a silica cake with a high solid content, 3 desintegrating the silica cake by subjecting it to chemical and mechanical operation in order to obtain a silica suspension with a high solid content and a low viscosity, able to be pumped and atomized, and 4) drying the desintegrated silica cake to obtain silica particles.

So as to measure the deagreggation ability of the thus obtained particles, Chevallier et al. an ultrasound test carried out on a silica suspension having a silica content of 4 %, the suspension being subjected to ultrasound for 420 sec.

The suspension with a high solid content and a low viscosity, able to be pumped and atomized resulting from the desintegrating of the silica exhibited an insufficient stability when stored. It tended to settle after a few hours storage.

At that time, commercialization of a stable suspension could not be planned. That is the reason why I was in charge of developing a silica suspension with a high solid content and low viscosity exhibiting an improved stability. In this project, I discovered that a more elaborate deagglomerating step provided a stable silica suspension having high silica content and a low viscosity.

So, the instant invention comprises after the separation step (B) a deagglomeration step (C) consisting in deagglomerating a silica cake so as to obtain an aqueous suspension of precipitated silica having a solid content between 10 and 40% by weight, a viscosity lower than 4.10^{-2} Pa.s at a shear rate of 50 s^{-1}, and a high stability in time, i.e. such a stability that the amount of silica present in the supernatant obtained after centrifuging the suspension at 7500 revolutions per minute represents more than 50% of the weight of the silica present in the suspension.

The following test was performed under my personal supervision.

Five suspensions (examples 1-5) of silica were prepared by the following steps :

(I) Five silica precipitation cakes were prepared according to the process disclosed in example 2 of the instant application. These Si/Al cakes correspond to cakes prepared according to the step (A) and (B) of the invention, and also according to the method disclosed in US 5,403,570.

(II) Each cake so obtained was submitted to a chemical crumbling with a sodium aluminate solution as disclosed in col.3, line50 of Chevallier et al followed by a deagglomeration step performed in a controlled manner with ultrasounds generated by a VIBRACELL BIOBLOCK (600 W) sonic transducer, with increasing deagglomeration times, so as to obtain suspensions 1 to 5.

The results obtained with 5 different deagglomeration times are shown in Table I, here below :

Table I

	Ex. 1	Ex. 2	Ex. 3	**Ex. 4**	**Ex. 5**
Deagglomeration time (min.)	0	5	10	**15**	**35**
Viscosity of the suspension (Pa.S)	$5{,}7.10^{-2}$	$2{,}9.10^{-2}$	$2{,}5.10^{-2}$	**$2{,}4.10^{-2}$**	**$1{,}2.10^{-2}$**
Amount of SiO_2 in the supernatant after centrifugation under 7500 rpm for 30 min.	9%	30%	41%	**52%**	**76%**

Example 1 illustrated the process disclosed in chevalier et al. Examples 4 and 5 illustrated the process of the instant invention.

It can be observed from example 2 that a 5 minute treatment with ultrasounds is sufficient for dramatically decreasing the viscosity of the suspension to

to half the inital value, thus leading to a suspension which is pumpable and spray-dryable according to Chevallier.

On the other hand, a suspension having a high stability, defined as a suspension exhibiting more than 50% of the initial weight of the silica present in the supernatant after centrifugating at 7500 rpm for 30 minutes, is only obtainable with a ultrasound treatment for at least 15 minutes.

The results presented here above are fully unexpected, since, in the field of silica suspensions, an intensive deagglomeration does not obviously lead to a long term stability, because of the possibility of reagglomeration phenomena.

Moreover, it has to be stressed that, because of the predicted difficulties regarding the evolution of a silica suspension, stabilized silica suspensions are usually obtained by adding stabilizing compounds such as dispersing agents. Therefore, it was not obvious that a stable silica suspension could be obtained only by a high mechanical deagglomeration, without chemical stabilization.

The undersigned Declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true ; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 2 day of March 2000

E. PRAT